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**MEMORANDUM** 

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FROM:	Andrew	W.ll	<u>.</u>			,	·
SUBJEC	T: Elimi	nation q	Precision	n Extrus	ion G.		
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Draft Letter

Richard Ziehm, President Precision Extrusion Company 720 E. Green Avenue Bensenville, IL 60106

Dear- Mr. Ziehm

I would like to thank you and Mr. Cramer for your assistance in the Department of Energy's investigations regarding past Atomic Energy Commission activities at your site. As I indicated based on the records we have identified and my discussions with you, we have concluded that there is little potential for residual contamination at your facility. As a result, we are closing our investigations with regard to the Precision Extrusion Company.

As per your request, enclosed are copies of the records we discussed in our telephone conversation of November 13, 1987. If you have any questions regarding this material please call me at (301) 353-5439.

sincerely

Andrew Wall o

Page No. 11/03/87

PRECISION

MIE FILE FRON T0 SUBJECT SITES BOX # 06/30/49 3.1CHO O'KOEFFE, 6./MOLAND, K. PRELIMINARY REPORT ON ALUMINUM-LITHIUM ALLOY PRODUCTION JOSEPH DIXON CRUCIBLE CO., HUC 2166 AL/LI Alloy for Coming No Exercise DETROIT GASKET AND MANUFACTURING CO. TRECISION EXTRUSION CO. 04/17/56 IL.20 GAUT, H. NOVAK, J. EXTRUSION OF URANIUM OXIDE ALUMINUM BILLETS IN BENSENVILLE, PRECISION EXTRUSION COMPANY, AND X22 2180 IL (PRECISION EXTRUSION COMPANY) (EXTRUDED FUEL PLATES FOR THE ARGONAUT REACTOR WHICH WAS TO BE EXHIBITED IN GENEVA) 09/30/50 HI.1 SCHLMAR, J. FOOTE, F. EXTRUSION OF EXPERIMENTAL FUEL CHANNEL TUBES (SECTIONS: PRECISION EXTRUSIONS, AIR FORCES 45/10 2240 ANE-4580, P.20) Al iuses only Cent find EXPERIMENTAL NETHODS PLANT ADRIAN LABOR AND TO PRECISION SCREENING --1/18/52

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11/13/67 He Knows work was done in

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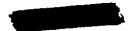
The early Years, Did alot of experimental

work.

Company President, Rochard 2 cm,

Richard Ziehm Pres

720 E. Green Ave Ben. 60106



## IV. Pouring Temperature

The pouring temperature for lithium, when the "bomb" technique is used, is approximately 200-250°C. When the aluminum and lithium are charged in the crucible as separate metals, the pouring temperature for the molten alloy is in the range 900-1000°C.

# V. Molds and Mold Washes

A number of mold materials and sizes have been tried. The best practical mold to date has been a 5° I.D. low-carbon steel mold with an alundum hot top. To date, several suitable mold washes have been found, although the most suitable has been 2r02 suspended in bugyl alcohol.

# VI. Billet Treatment

After removal from the molds the billets are prepared for extrusion in the following manner:

- 1. The hot top section and a slice from the bottom are out from the billets and are used for analytical camples.
- 2. The billet surfaces are machined clean of any porosity, usually requiring a cut of from  $1/4^n$  to  $1/2^n$  from the diameter on  $5^n$  billets.
- If billets are to be shipped to an outside extrusion plant, the 5" billets are sent to Detroit Gasket and Hamufacturing Company and 3" billets to the Precision Extrusion Company, Bensonville, Illinois. Before shipping they are wrapped and boosd for protection.

The program for developing a means of producing an alloy of approximately 3 to 4 weight percent lithium in aluminum is mearing completion. During the course of this program emough alloy was cast to provide more than 2000 extruded and machined slugs to Hanford for radiation purposes.

Although production responsibilities of the alloy have been transferred to Hanford, it is not felt that sufficient work has been done on the subjects of mold material, mold temperature, and type of wash to insure consistent billets of sufficiently good surface to allow their extrusion without machining. Billet diameter is critical as good 3" billets have been easily reproduced whereas 5" billets have not, generally requiring from and eighth inch to over one-half inch of metal to be machined from their outside diameters to remove surface defects before extrusion.



#### Crucibles

During the course of the program, crucibles having wall thicknesses up to 2" have been machined from graphite of various grades including AGHT, a more dense and higher purity grade than usually available, and all failed by cracking through the side walls and bottoms as shown in Figures 1 and 1-a. This is true only for 5" castings, however, as the graphite crucibles were successful when used for 3" billets. Excellent results have been obtained by using a SiC2 clay-bonded graphite, unglased crucible, #60, made by the Joseph Dixon Crucible Company, Jersey City, New Jersey. Other suppliers make a grucible of similar type but most of them use a borosilicate glass which badly contaminates the resultant alloy with highly undesirable boron. Bixon crucibles can be used for eight to ten melts, and although they are still in excellent condition generally, the stopper rod boles are badly enlarged because of cleaning damage. It is sometimes necessary to drill a little larger hole in the crucibles to clean them, and it is always necessary to reseat the crucible with a 60° reasor, which matches the taper machined on the ends of the stopper rods. A 5/8" hole is usually drilled in a new erucible for the first melt. MgO crucibles, including a high purity A-312 crucible, proved unsatisfactory by cracking and by producing a severe reaction with the alloy as shown in Figures 2 and 2-a. Alundum and BeO crucibles have and can be used but due to their high cost, short life, and the toxicity hazard of BeO, plus the fact that they are difficult to elean and slow down the melting cycle, makes them impractical. The melting cycle is slower as all refractory materials require longer heating periods due to susceptibility to thermal shock.

## Stopper Rods

A number of stopper rods have also been tried, namely: high fired BeO, alundum, SiC<sub>2</sub>, AGHT graphite and graphite of questionable history. Difficulty had been experienced with stopper rods fracturing during melting and shifting of the charge. This was true of BeO, alundum, questionable graphite, and SiC<sub>2</sub> rods, necessitating their abandomment. The SiC<sub>2</sub> rods, nade from globars, failed since the rods break at the junction of the globar element and the end element when the rod is pulled to make a casting. This failure, peculiar to this rod, is due to the method of manufacture of the globar. The AGHT graphite rods give excellent service and it was found they could be used for at least five to ten heats, if cleaned between melts, by taking a slight out from the seating end of the rod in a lathe. Rods ranged in size from 3/4" to ½" in diameter and from 18" to 20" long. The AGHT rods can be used as small as 3/4" in diameter, with no failure, but it is well to start with at least a 1" rod, as the cleaning operation removes a little each time from the diameter. (Figure 3 shows type of failure in graphite rods of questionable history.)



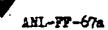
#### <u> Molda</u>

Molds have been made of a number of different materials, namely low carbon steel, porous alundum, pyrex tubing and graphite of unknown type and density. Variable wall thicknesses of from 12" to 11/16" have been tried with graphite molds, to influence the solidification rates, with consistently poor results in regard to surface quality, with or without the use of mold washes. Excellent billets have been cast in alundum tubes which are porous and allow gases to pass through, and which also allow slow cooling rates. Alundum tubing is expensive and can be used only once, as it cracks upon solidification of the alloy, and must be encased in graphite for strongth and to prevent loss from leakage in case the alundum cracks before the metal solidifies. Pyrex glass tubes produce good billets with excellent surfaces; however, the glass reacts slightly with the billet surface leaving a hard glassy layer which spalls off, endangering personnel. Pyrex tubing is also expensive, can be used only once, and requires the same handling as alundum. Cylindrical low carbon steel molds have produced billets having the best consistent surface qualities. Although the first billets cast in the latter molds did not have excellent surface qualities, it was found that the surface porosity did not penetrate the billsts deeply and that the surfaces could be cleaned up by machining sometimes less than 1/8" from their dismeters. Although all molds tried are heated to some degree at their upper hot topped ends, because they enter the fringes of the field produced by the induction coil used for vacuum melting, three billets were cast in a low carbon steel mold intentionally heated with an external furnace to 265°C. Two of these were excellent, the other melted from scrap alloy was of poor quality, but it is not known that the poor quality was related to the mold temperature. It is felt that the use of heated molds should be further explored. The external heater used on these molds was a resistance wound furnace, encased in cheet metal, with the leads being passed through the bottom of the induction furnace by means of special vacuum connectors, soldsred in the furnace bottom, and the leads then scaled with a vacuus scaling wax. (Figure 4 is a picture of heater and type of connectors used.) The graphite and low carbon steel molds, the only molds hot topped, are not topped by placing a piece of alumdum tubing 5" to 6" long in a counter bored section inside the top of each mold. (Figures 5 and 5-a are pictures of het topped low carbon steel mold.) Cleaning of the steel mold in dilute HCl is necessary, at least after every third casting. It is felt that an attempt should be made to use a sand mold, possibly baked to strengthen and remove moisture, and encased in a graphite sleeve for further protection. This mold, made from ordinary foundry sand, might possibly simulate the alundum molds and sould be expendable or broken up to be remolded.

### Mold Washes

A number of mold washes have been used in connection with mold tests. Very poor results were obtained with  $2rO_2$  in shellar, high purity MgO, and PbO<sub>2</sub>. The better washes, on any mold, were  $Al_2O_3$ ,  $2rO_2$ , and a wash used







commercially composed of MagSiO3, CaCO3 and water applied to the steel mold heated to 235°C. Unless specified otherwise, all of the above were suspended in butyl alcohol. As stated before, use of a wash is not necessary, but one may be used if billst removal becomes difficult.

# Charging Technique

If not protected from the atmosphere when melted, lithium mill react and burn, so it was necessary, in some way, to get the lithium in solution with the aluminum while still protecting the lithium. The following is a regume of the first attempts to make the alloy. The first seven experimental melts were made by melting the aluminum under a LiGl flux, in aluminum erucibles, and by adding the lithium either encased in aluminum tubing or by holding it under the aluminum with an iron plunger until it melted. Although it was possible to make the alloy using this method, the cast metal contained many inclusions of the deliquessment LiGl flux which picked up atmospheric moisture, and it was decided to attempt to use vacuum melting and casting techniques to lower the total H content, and to lessen metallic contamination.

One attempt was made to wantum melt by placing the aluminum and lithium in an alundum crucible and to try to cast by means of an alundum stopper rod. The melt was unsuccessful as the lithium burned and reacted with the crucible. At this point it was decided to machine some aluminum bombs to be filled with lithium in the correct proportion to form the desired alloy upon melting. The lithium sealed in the aluminum bombs would be protected from evaporation and oxidation during the period it takes to melt the aluminum, and it was also felt this method would help to disperse the lithium more uniformly in the melt and minimize the possibility of segregation. The 25 aluminum bombs were machined from 2" diameter rod stock, 11" long with a hole 0.920" in diameter by 10" deep, so that the filling of the bosh to the top of the machined hole with molten lithium would automatically combine the two elements at four weight percent lithium. A 2" threaded cap seals the topof the bosh (Figure 6, sketch of bosh). Of course, it was first necessary to melt the lithium before pouring it into the aluminum bombs. This was first accomplished through the use of Dow Corning Corporation silicone fluids of the series 200-500, as it was found lithium could be protected from atmospheric combination through the use of these fluids. By simply wetting the pieces of metal with the fluid they were melted in open Armoo iron pots, heated by bunsen burners, and then poured over into the bombs. In order to speed up the operation, and to stockpile a few boxbs, a new lithium furnace was built consisting of an Armoo iron crucible and stopper rod, scaled with a lid, and heated by a resistance wound furnace as shown in Figures 7, 7-a, and 8. A tank of argon gas is connected to a pipe fitting in the lid of the furnace to provide an inert atmosphere and to supply pressure in order to force the molten metal, which does not flow by gravity alone, to flow into the bombs when the stopper rod is lifted. With the aluminum bombs preheated to 100°C and assembled into the filling device, the lithium, at 225-250°C, is poured into the bombs and the threaded cap, lubricated with a small amount of





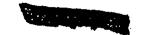
DC-500 silicone fluid, is sorewed into place. Harm dry sand is kept in readiness to extinguish small fires resulting from spills that might occur during this operation, and it is a good policy to provide a steel tray beneath the equipment to prevent the lithium from contacting the coment floor. Although this crucible will hold sufficient lithium to fill about 40 bombs, holding approximately 65 grass each, usually only 20-25 bombs are filled at a time for safety precautions. Cleaning of the crucible, of oxide and metal left behind, is accomplished, between malts, by taking it from the building without the lid and filling it quickly with water. This is done by standing behind a shelter and using a water hose. The hole in the bottom of the crucible is first plugged so that the water doesn't leak out, making it possible to fill the crucible. The hose is usually kept on the crucible until all reaction ceases. This is a rather besardous method but is the only quick procedure found.

Toward the end of the program, after satisfactory crucibles and stopper rods had been obtained, two eastings were again tried without filling aluminum bombs with lithium, the regular practice, by adding the aluminum and lithium separately in solid form and vacuum melting at a pressure of approximately 300 microns. There was no reaction with the Dixon crucible, such as was encountered with all other crucibles, and only 0.3% lithium was lost in these billets, indicating that this technique was satisfactory and could be immediately adopted.

## Melting Procedure and Vacuum Rouisment

The operation of the micerta tube vacuum furnace may best be described by listing the steps necessary to make a casting. Assuming that the cycle begins with the furnace empty and the top and bottom plates removed, the steps in the furnace operation are as follows:

- 1. The mold is placed on the furnace bottom plate.
- 2. The crucible is placed above the mold, and the entire assembly is elevated into the furnece.
- 3. The bottom plate is bolted to the bottom of the furnace.
- 4. The charge is top loaded into the crucible around the stopper rod.
- 5. The crucible cover and top insulating bricks are put in place.
- 5. The furnace top plates are put into position, the end of the steel stopper rod extension being pushed through the Wilson seal in the auxiliary top plate.
- 7. The furnace is evacuated, or an inert atmosphere is admitted if desired.



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- 8. The charge is melted and brought to the proper casting temperature, 900-1000°C. The temperature is read through the glass window in the auxiliary top plate with an optical pyrometer.
- 9. The molten charge is allowed to run into the mold by pulling up on the stopper rod.
- 10. After the crucible has cooled to a black or dull red temperature, argon is admitted to the furnace, and the top plates are removed.
- 11. The stopper rod, top bricks and cover are removed from the crucible.
- 12. The bottom plate is unbolted and the plate, mold, and crucible are lowered out of the furnace. The furnace is now ready for reassembling for making another easting. Figure 10 shows furnace bottom with mold and bester.

The current used for heating the vacuum induction furnaces is 3000 cycle, 400 volt A.C. and is supplied by a 50 KW air cooled Westinghouse Motor-Generator set. The capacitance for the high frequency circuit is supplied by a bank of five multi-tap water cooled capacitors. In operating a furnace, the correct amount of capacitance is placed in the circuit to give a power factor as near to unity as possible. The high frequency current is carried to the furnaces by a bus-bar system. Each furnace is equipped with knife switches so that it can be easily connected to or disconnected from the main bus line. By means of a control panel any power from a few kilowatts to the maximum output of the generator can be obtained quickly and easily. The generator and capacitor bank are fully protected by overload relays.

The furnaces are equipped with a manifold type vacuum system. The main vacuum line and the lines to the furnaces are made of extra heavy 2° steel pipe. All threaded joints are packed with a litharge and glycerin mixture. Each furnace is equipped with its own valve and dial type vacuum gauge. A main line valve is located at the pump, and a McLeod Gauge is placed in the line for obtaining accurate pressure readings. Each furnace and the main line have Protect-O-Notor Filters installed. Each furnace has a bleed-off valve so that the vacuum in any unit can be broken without allowing air to enter the main line. Also, these bleed-off valves are used for admitting inert atmospheres into the furnace.

The vacuum pump now in use is a Kimmey 105 subic foot per minute, water-cooled pump. This pump is capable of producing a vacuum of about 15 microns pressure. A vacuum of 200-1000 microns is obtainable in the malting furnaces.

### Analysis

Good adherence to desired analysis has been obtained, during the program, as is shown in the tables. Hentes M46 and M56 which are high in lithium are believed to be due to a leaking stopper rod which allowed some lithium to run into the mold to solidify before the remainder of the charge was poured. Some of the billets included on these tables were made by melting scrap obtained from previous billets, extrusion skulls, etc., and after remaiting it was found that only 0.15 lithium had been lost. The percentage of



major impurities such as iron, copper and boron have been lowered by a large factor since vacuum malting was adopted.

# Extrusion

Extrusion of the alloy has presented no problem in either the 3" or 5" billets. After machining, to clean up their surfaces, the billets were taken to an outside concern for extrusion, the 3ª billets having been extruded by the Precision Extrusion Company, Bensonville, Illinois; and the 5" billets by the Detroit Casket and Manufacturing Company, Detroit, Michigan. Host of the alloy has been extruded from 5" billets and the data is as follows: preheat time takes about 20-30 minutes to reach the lowest satisfactory extrusion temperature which is between 210-260°C. The temperature is considerably higher, of course, in the preheat furnace itself, a muffle type natural mas-fired furnace. We have found that rods can be extruded without using a lubricant on the die, providing the die is polished after about ten extrusions. The lubricant, when used, is graphite in oil. Extrusion pressures for 5" billets range between 3000-4000 pai ram pressure. This is on a 1000 ton press with a 5 3/4" diameter container and a 27.165" diameter ram. Extrusion temperature for the 3" billets is about the same as for the 5", possibly a little lower, and they push easily on a 250 ton press. Rods are extruded through a 1.375° diameter bell mouth type die, and upon cooling sirrink to approximately 1.370°. They are then straightened on a stretch streightener, this operation reducing the diameter again to approximately 1.363". The rods are then out into 22" lengths and returned to our machine shop where they are cut to slug length, machined to 1.360" to 1.350" diameter, packed in aluminum cans for protection and shipped to Hanford, Washington, The die size should be at least .010 larger to allow more metal for machining purposes.

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30 September 1950

Program 7.7.5

Tot

Frank Foots

From:

J. P. Schumar, R. A. Noland

SED<del>UMIT MIDM</del>IATION

Ret

EXTRUSION OF EXPERIMENTAL FUEL CHANNEL TUBES (Section: ANI-4580, p.20)

Fuel channel tubes and the corresponding fuel jackets have been extruded at Precision Extrusions and at the Air Forces Experimental Methods Plant at Adrian, Michigan. A contract has been let with the latter organization, and we are now working with them on three and four tube webbed clusters to be extruded from 2S and 63S aluminum alloys. A stockpile of extrusion billets is now being set up to provide for a dependable supply of material.

Distributions

1. Program 7.7.5

2. ANL-FF-239



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